

Effect of Temperature on the Extraction of Sicilian and Domestic Sumacs^{*†}

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The temperature at which a tanning material is leached is important both from the standpoint of amount of material extracted and from that of the tanning quality of the extract. The present investigation is concerned primarily with the quantity of soluble matter extracted at 100° C. and at various lower temperatures. The tanning quality of extracts produced at the different temperatures has been considered only to a limited extent, although this is just as important as quantity of extract.

It is commonly believed that there is an optimum temperature for the extraction of each raw tanning material. This view for example, has been expressed as follows by Procter:⁹ "Whether in the manufacture of extracts or for direct use in the tannery the temperature at which tanning materials are extracted is of prime importance. It is a common mistake to assume that the largest amount of tannin is extracted by boiling. A. N. Palmer has pointed out that this is by no means the case but that each material has an optimum temperature of extraction at which more tannin is extracted than at any other." There has not been agreement, however, as to the optimum temperature for extracting sumac.

Sumac normally is leached at temperatures below 100° C., but Eitner³ in 1895 leached six tanning materials, including sumac, at higher temperatures. These were 100°, 121°, 144°, and 159° C., and were obtained by raising the pressure to 1, 2, 4, and 6 atmospheres, respectively. He obtained 22.8 per cent tannin at 100°, 22.7 per cent at 121°, 11.3 per cent at 144°, and 8.9 per cent at 159° C., indicating that there may be a maximum extraction of tannin at some point between 100° and 121° C. Non tannin increased rapidly as the temperature was raised above 100° C., and reached a maximum at 144° C. His highest soluble solids value was obtained at 121° C.

Parker and Procter⁸ in 1895 found that a maximum amount of both soluble solids and tannin were extracted from sumac at 50° to 60° C. Their work will be discussed briefly after our data are presented, since their results are frequently cited, for example, by Harvey⁶ and Gnam.⁵

In 1906 Veitch and Hurt¹⁵ compared several extractors and temperatures

^{*} This paper reports the results of one phase of the cooperative investigations of American sumac as a commercial source of tannin by the Bureau of Agricultural and Industrial Chemistry and Bureau of Plant Industry, Soils, and Agricultural Engineering, Agricultural Research Administration, and Soil Conservation Service, U. S. Department of Agriculture.

[†] Presented at the Forty-Third Annual Meeting of the American Leather Chemists Association, Edgewater Beach Hotel, Chicago, Ill., June 19, 1947.

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for extracting sumac for analysis. They obtained highest results when boiling water was used during the latter part of the extraction.

Veitch¹⁴ in 1908 made the following statement: "It has been the opinion among tannery chemists, based on the work of Seymour-Jones, Palmer, and Parker and Procter of England, that most tanning materials are best extracted at temperatures below boiling; thus sumac is supposed to yield the highest results by extracting below 60° C. The work done in the Bureau of Chemistry makes this opinion no longer tenable as the highest results have been obtained at from 60° to 90° C." He attributed the difference in optimum temperatures to an improved method, which gave a more nearly complete extraction of the sumac. His soluble solids and tannin values were all appreciably higher than those of Parker and Procter.

A study of the extraction of sumac for analysis was made by Stather¹² in 1933. He extracted four samples at each of the temperatures 20°, 40°, 60°, 80°, and 100° C., and found a maximum yield of soluble matter and tannin at 100° C. He concluded that the opinion that sumac tannin is decomposed on long heating with water, as in the Grasser-Allen extractor, is not true.

Inconsistent results for tannin in commercial sumac samples by different analysts were attributed by Simoncini¹¹ in 1937 to incomplete extraction of the material. He concluded that the sample must be finely ground and then extracted at an average temperature of 95° to 100° C. for 8 hours or longer. He also stated that the extract solution might be concentrated, if necessary, over direct fire to the required volume.

Stather and Lauffmann¹³ in 1938 examined the extracts prepared by leaching several tanning materials at 20° and at 100° C. They concluded that hot extraction of sumac had no bad effect on the extract.

Description of Samples and Methods

Previous work on the effect of temperature on the extraction of sumac apparently has all been done on *Rhus coriaria*. In the present work it was desired especially to study the behavior of the three common American species and to compare their behavior in regard to temperature of extraction with that of *R. coriaria*. Accordingly, three samples were obtained of each of the following species: *Rhus copallina* L. (dwarf sumac), *R. coriaria* L. (Sicilian sumac), *R. glabra* L. (white sumac) and *R. typhina* Torn. (stag-horn sumac). For convenience the samples were labeled by a number preceded by the first letter of the species name, except for *R. coriaria* for which the first letter of the commercial name was chosen. The sample of highest tannin content for each species was numbered 1 and that of lowest tannin content, 3.

All samples of domestic sumac were dried under shelter in the air at atmospheric temperatures except as noted, and were then ground in a Wiley mill to pass a 2 mm. screen. The *Rhus coriaria* was used as ground in Sicily.

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A brief description of each sample follows:

Sample C1. *Rhus copallina* leaves collected in 1945 from cultivated plants growing at the Bureau of Plant Industry, Soils, and Agricultural Engineering, East Farm, Beltsville, Maryland.

Sample C2. *R. copallina* leaf and leaflet mixture; about 58 per cent leaves and 42 per cent leaflets. Collected in 1943 at Beltsville, Maryland. This sample was used in previous work,⁷ Table I, and by Clarke, Mann, and Rogers,² Sample B.

TABLE I
ANALYSES OF SAMPLES BY AMERICAN LEATHER
CHEMISTS ASSOCIATION METHOD

Sample No.	Species of Rhus	Part of Plant	Insolubles in Extractive	Soluble Solids	Non Tannin	Tannin
			%	%	%	%
C1	<i>R. copallina</i>	leaves	1.6	56.4	22.3	34.1
C2	"	leaflets	1.7	52.0	18.8	33.2
C3	"	leaves	1.5	47.7	20.4	27.3
S1	<i>R. coriaria</i>	leaves*	3.0	53.1	20.4	32.7
S2	"	leaves	1.6	50.7	19.6	31.1
S3	"	leaves	1.9	50.0	19.9	30.1
G1	<i>R. glabra</i>	leaflets	1.6	48.9	20.1	28.8
G2	"	leaves	1.7	48.3	21.4	26.9
G3	"	leaflets	1.6	48.2	24.0	24.2
T1	<i>R. typhina</i>	leaflets	1.6	49.7	22.5	27.2
T2	"	leaflets	2.1	48.2	22.8	25.4
T3	"	leaves	2.0	43.2	20.0	23.2

*Commercial preparation, essentially leaves.

Sample C3. *R. copallina* leaves collected in 1943 near Wells Tannery, Pennsylvania.

Sample S1. *R. coriaria* leaf material. Commercially ground Sicilian leaf imported in 1939. A report by the Microanalytical Division, Food and Drug Administration, on this and the following sample (S2) read as follows: "Microscopic examination shows these samples to consist essentially of commercially pure ground Sicilian sumac made from the leaves of *R. coriaria* L. or some closely related species. There is present a trace of other plant tissues, mainly some particles of cereal chaff resembling oat. No evidence of the presence of lentiscus or tamarisk found."

Sample S2. *R. coriaria*. Similar to sample S1 but imported in 1937.

Sample S3. *R. coriaria*. Although obtained from a tannery in 1944 the sample was from a shipment imported in 1939. It is possible that samples S1 and S3 were from different bags of the same importation.

Sample G1. *R. glabra* leaflets. Collected in 1943 near Hagerstown, Mary-

TABLE II
EFFECT OF TEMPERATURE OF EXTRACTION ON THE AMOUNT OF EXTRACTIVES
OBTAINED FROM THREE SAMPLES OF *Rhus copallina* LEAVES

Sample No.	Extraction Temperature	Insolubles in Extractive	Soluble Solids	Non Tannin	Tannin	Purity of Extractive	Reducing Sugar	Non-reducing Sugar	Total Sugar
	° C.	%	%	%	%	%	%	%	%
C1	2°	0.2	31.4	19.1	12.3	39	3.5	3.3	6.8
	10°	0.2	38.1	20.2	17.9	47	3.9	2.2	6.1
	20°	0.1	43.1	19.5	23.6	55	4.1	2.0	6.1
	30°	0.3	50.2	20.3	29.9	60	4.2	2.0	6.2
	40°	0.3	52.3	20.3	32.0	61	4.5	1.7	6.2
	50°	0.4	50.9	20.6	30.3	60	5.0	0.8	5.8
	60°	0.7	50.9	21.0	29.9	59	4.7	1.4	6.1
	70°	0.7	53.1	21.2	31.9	60	5.0	1.2	6.2
	80°	1.2	54.8	21.5	33.3	61	4.7	1.6	6.3
	90°	2.0	57.3	22.6	34.7	61	4.8	2.0	6.8
	100°	2.4	58.8	23.3	35.5	60	4.1	2.9	7.0
C2	2°	0.3	24.0	14.3	9.7	40	2.3	1.5	3.8
	10°	0.4	32.8	16.5	16.3	50	2.7	1.5	4.2
	20°	0.2	38.8	16.1	22.7	59	2.9	1.3	4.2
	30°	0.7	44.5	17.0	27.5	62	3.2	1.3	4.5
	40°	0.4	46.3	17.1	29.2	63	2.8	1.8	4.6
	50°	0.6	46.0	17.1	28.9	63	3.1	1.4	4.5
	60°	0.8	46.5	17.6	28.9	62	3.0	1.4	4.4
	70°	1.0	48.0	17.6	30.4	63	2.9	1.6	4.5
	80°	1.6	49.7	17.9	31.8	64	3.0	1.7	4.7
	90°	2.3	51.5	18.4	33.1	64	2.9	2.3	5.2
	100°	3.0	52.8	19.4	33.4	63	2.8	2.4	5.2
C3	2°	0.1	27.5	17.3	10.2	37	3.7	1.4	5.1
	10°	0.1	32.7	18.0	14.7	45	3.8	1.4	5.2
	20°	0.2	37.2	17.6	19.6	53	3.8	1.3	5.1
	30°	0.2	41.4	18.4	23.0	56	4.2	1.1	5.3
	40°	0.1	43.3	18.7	24.6	57	3.9	1.4	5.3
	50°	0.3	42.3	19.0	23.3	55	4.1	1.3	5.4
	60°	0.5	42.7	19.7	23.0	54	4.0	1.3	5.3
	70°	0.6	44.0	19.3	24.7	56	4.0	1.4	5.4
	80°	1.4	45.5	19.4	26.1	57	3.9	1.7	5.6
	90°	2.0	47.5	20.3	27.2	57	4.1	1.8	5.9
	100°	2.8	49.1	21.2	27.9	57	4.1	2.0	6.1

land, and air-elutriated to remove petiole-rachises (See Clarke, Mann and Rogers,² Sample F; Luvisi and Clarke,⁷ Table III).

Sample G2. *R. glabra* leaves collected on September 22, 1944, from cultivated plants growing at East Farm, Beltsville, Maryland. Dried in trays in moving air at 40° C.; dried in less than one day.

Sample G3. *R. glabra* leaflets, hand separated from leaves. Collected on August 21 and 24, 1944, from wild plants at Wyndmoor, Pennsylvania.

TABLE III
EFFECT OF TEMPERATURE OF EXTRACTION ON THE AMOUNT OF EXTRACTIVES
OBTAINED FROM THREE SAMPLES OF *Rhus coriaria* LEAVES

Sample No.	Extraction Temperature	Insolubles in Ex-tractive	Soluble Solids	Non Tannin	Tannin	Purity of Extractive	Reducing Sugar	Non-reducing Sugar	Total Sugar
	C°	%	%	%	%	%	%	%	%
S1	2°	0.5	42.2	17.8	24.4	58	3.0	1.7	4.7
	10°	0.3	47.6	18.9	28.7	60	2.3	2.3	4.6
	20°	0.5	48.8	17.9	30.9	63	3.4	1.3	4.7
	30°	0.7	49.7	18.2	31.5	63	3.6	1.0	4.6
	40°	0.6	48.7	18.2	30.5	63	3.5	1.2	4.7
	50°	1.2	48.4	18.3	30.1	62	3.6	1.1	4.7
	60°	1.2	50.2	18.5	31.7	63	3.7	1.1	4.8
	70°	1.6	50.8	18.6	32.2	63	3.7	1.0	4.7
	80°	2.1	52.0	19.2	32.8	63	3.5	1.1	4.6
	90°	2.7	53.7	20.3	33.4	62	3.7	1.3	5.0
	100°	3.8	55.3	21.3	34.0	61	3.5	1.5	5.0
S2	2°	0.1	40.0	17.6	22.4	56	2.9	1.6	4.5
	10°	0.3	45.5	17.0	28.5	63	3.2	1.4	4.6
	20°	0.2	46.8	17.8	29.0	62	3.3	0.9	4.2
	30°	0.5	47.7	18.5	29.2	61	3.3	1.2	4.5
	40°	0.6	46.5	18.3	28.2	61	3.4	1.2	4.6
	50°	1.1	46.7	18.5	28.2	60	3.5	1.1	4.6
	60°	1.2	47.8	18.8	29.0	61	3.5	1.1	4.6
	70°	1.9	48.8	18.7	30.1	62	3.6	1.0	4.6
	80°	2.3	49.6	19.2	30.4	61	3.4	1.2	4.6
	90°	2.8	51.4	20.4	31.0	60	3.5	1.3	4.8
	100°	3.6	52.1	20.9	31.2	60	3.4	1.4	4.8
S3	2°	0.3	40.0	17.7	22.3	56	2.9	1.3	4.2
	10°	0.3	45.1	17.5	27.6	61	3.0	1.3	4.3
	20°	0.3	46.1	17.8	28.3	61	3.2	1.0	4.2
	30°	0.1	47.2	18.6	28.6	61	3.3	0.8	4.1
	40°	0.6	46.2	18.5	27.7	60	3.3	0.8	4.1
	50°	1.1	46.5	18.4	28.1	60	3.4	0.9	4.3
	60°	1.1	47.5	18.7	28.8	61	3.5	0.8	4.3
	70°	1.7	48.0	18.7	29.3	61	3.3	0.8	4.1
	80°	2.3	49.2	19.3	29.9	61	3.2	0.9	4.1
	90°	2.6	50.9	20.2	30.7	60	3.4	1.1	4.5
	100°	3.8	51.4	20.5	30.9	60	3.2	1.3	4.5

Sample T1. *R. typhina* leaflets, hand picked on August 2, 1944, from wild plants at Wyndmoor, Pennsylvania. (See Luvisi and Clarke,⁷ Table IV.)

Sample T2. *R. typhina* leaflets, hand picked on August 21 and 24, 1944, from wild plants at Wyndmoor, Pennsylvania.

Sample T3. *R. typhina* leaves collected on September 22, 1944, from cultivated plants growing at East Farm, Beltsville, Maryland. Dried in trays in moving air at 40° C.; dried in less than one day.

TABLE IV
EFFECT OF TEMPERATURE OF EXTRACTION ON THE AMOUNT OF EXTRACTIVES
OBTAINED FROM THREE SAMPLES OF *Rhus glabra* LEAVES

Sample No.	Extraction Temperature	Insolubles in Ex-tractive	Soluble Solids	Non Tannin	Tannin	Purity of Extractive	Reducing Sugar	Non-reducing Sugar	Total Sugar
	° C.	%	%	%	%	%	%	%	%
G1	2°	0.3	29.3	16.5	12.8	44	3.4	1.6	5.0
	10°	0.3	37.2	17.7	19.5	52	3.5	1.7	5.2
	20°	0.1	42.5	17.5	25.0	59	3.7	1.5	5.2
	30°	0.1	44.3	18.3	26.0	59	4.1	1.2	5.3
	40°	0.4	44.0	18.4	25.6	58	3.8	1.4	5.2
	50°	0.5	43.7	18.7	25.0	57	3.9	1.5	5.4
	60°	0.5	44.9	19.1	25.8	57	4.0	1.1	5.1
	70°	1.2	45.6	18.9	26.7	59	4.0	1.2	5.2
	80°	1.3	46.6	19.0	27.6	59	3.7	1.5	5.2
	90°	2.5	48.6	20.4	28.2	58	3.8	1.7	5.5
	100°	3.4	50.2	21.5	28.7	57	3.6	2.2	5.8
G2	2°	0.2	31.4	18.7	12.7	40	2.6	4.7	7.3
	10°	0.3	38.0	19.2	18.8	49	2.9	4.4	7.3
	20°	0.2	42.5	18.6	23.9	56	3.4	4.0	7.4
	30°	0.2	43.8	19.4	24.4	56	4.5	2.8	7.3
	40°	0.7	43.7	19.4	24.3	56	4.2	3.1	7.3
	50°	0.6	42.7	19.4	23.3	55	5.3	2.0	7.3
	60°	0.9	43.3	19.7	23.6	55	5.5	1.6	7.1
	70°	0.9	44.6	19.9	24.7	55	5.6	1.8	7.4
	80°	1.4	45.6	20.1	25.5	56	4.9	2.7	7.6
	90°	1.8	47.9	21.2	26.7	56	4.9	3.2	8.1
	100°	2.5	50.0	22.4	27.6	55	4.3	3.9	8.2
G3	2°	0.2	33.2	20.5	12.7	38	2.2	4.6	6.8
	10°	0.2	40.0	21.2	18.8	47	2.5	4.3	6.8
	20°	0.3	44.1	21.2	22.9	52	3.1	3.8	6.9
	30°	0.3	45.2	21.6	23.6	52	4.7	2.4	7.1
	40°	0.8	44.8	21.8	23.0	51	4.3	2.9	7.2
	50°	0.7	42.5	22.0	20.5	48	5.4	1.7	7.1
	60°	0.8	43.7	22.4	21.3	49	5.6	1.5	7.1
	70°	1.0	44.4	22.5	21.9	49	5.5	1.5	7.0
	80°	1.4	46.3	23.1	23.2	50	5.2	2.1	7.3
	90°	2.4	48.7	24.4	24.3	50	5.0	2.6	7.6
	100°	2.9	51.1	25.6	25.5	50	5.0	2.9	7.9

An analysis of each sample by the Official method¹ is given in Table I. These tannin values were used for calculating the quantity of material to be used in the temperature studies. It was anticipated that at the lower extraction temperatures less tannin would be extracted than by the Official method. To follow the method it would have been necessary to make preliminary analyses and to use different quantities of sample at each extraction temperature. This would have introduced another variable, however, for a small quantity of material is leached more efficiently by a given volume of water

than a large quantity. The method adopted was to choose a quantity of each material calculated to yield solutions containing 4.2 grams of tannin per liter as determined by the Official method and to use this same quantity at all temperatures. It was expected that at the higher temperatures the tannin concentration would be near the upper limit¹ of 4.25 and at the lower temperatures near or even below the lower limit of 3.75. Soluble solids values at the various temperatures, however, should be strictly comparable.

Extractions were made in a modified Reed-Churchill extractor⁴ at 2° C. and at 10°-intervals from 10° to 100° C. The extractor was not equipped with a cooling unit, but water from a cooling bath was circulated through it for work at 10° C. Extractions at 2° and 20° C. were made in rooms regulated to these temperatures. At temperatures at 30° C. or above there was a deviation of less than 0.1 degree from that desired, and at 2°, 10° and 20° C. the deviation was not more than 0.5 degree throughout the extraction period.

An extraction was made by weighing the required amount of sumac, transferring it dry to the extractor, wetting it with water at the desired temperature, adjusting the water level so the sumac was just covered, and then leaching so as to obtain two liters of extractive in seven hours.

Some difficulty due to clogging of the extractor was experienced with *Rhus coriaria* at all temperatures and with *R. typhina* at 90° and 100° C. To minimize this, a layer of washed quartz sand about one-half inch deep was spread over the cotton in the bottom of the extractor before adding the sumac.

At the end of the extraction period, all solutions except those obtained at 90° and 100° C. were heated to 80° C., then cooled and analyzed by the Official method.¹

All non tannin determinations were made with 46 grams of wet chromed hide powder per 200 ml. of total solids solution except for extracts obtained at 2° and 10° C. For the latter, the hide powder per determination was reduced in proportion to the expected tannin concentration.

In addition to the usual tannin analyses, determinations were made of reducing and total sugars in all solutions and of pH values of all extractives except those obtained at 40°, 50°, 70°, and 80° C.

Sheepskin skivers were tanned by the method of Riethof¹⁰ with the total solids solutions obtained at 30°, 60° and 90° C. Colors of the skivers and of certain of the soluble solids solutions were evaluated by measuring percentage reflectance or transmission of light at wave lengths of 436, 546 and approximately 620 millimicrons.

Results

Analyses of the extracts obtained by leaching the various samples at the different temperatures are given in Tables II to V, inclusive.

A few of the tannin and non tannin values may be slightly in error because

TABLE V
EFFECT OF TEMPERATURE OF EXTRACTION ON THE AMOUNT OF EXTRACTIVES
OBTAINED FROM THREE SAMPLES OF *Rhus typhina* LEAVES

Sample No.	Extraction Temperature	Insolubles in Extractive	Soluble Solids	Non Tannin	Tannin	Purity of Extractive	Reducing Sugar	Non-reducing Sugar	Total Sugar
	° C.	%	%	%	%	%	%	%	%
T1	2°	0.3	31.2	18.2	13.0	42	2.4	3.2	5.6
	10°	0.3	39.2	19.6	19.6	50	2.8	3.0	5.8
	20°	0.8	43.5	19.4	24.1	55	3.5	2.5	6.0
	30°	0.4	44.9	20.1	24.8	55	4.6	1.2	5.8
	40°	0.5	45.0	20.1	24.9	55	4.6	1.4	6.0
	50°	0.8	43.6	20.4	23.2	53	4.9	1.2	6.1
	60°	0.8	45.3	21.2	24.1	53	4.8	1.0	5.8
	70°	1.1	46.4	21.7	24.7	53	4.8	1.0	5.8
	80°	1.2	48.3	22.4	25.9	54	4.7	1.3	6.0
	90°	3.0	50.4	24.1	26.3	52	4.7	1.5	6.2
	100°	2.8	53.7	26.6	27.1	50	4.0	2.6	6.6
T2	2°	0.1	29.2	18.2	11.0	38	2.6	3.8	6.4
	10°	0.3	39.4	19.8	19.6	50	3.2	3.6	6.8
	20°	0.4	42.1	19.9	22.2	53	3.9	2.9	6.8
	30°	0.4	43.8	20.6	23.1	53	5.1	2.0	7.1
	40°	0.3	43.2	20.4	22.8	53	5.1	1.8	6.9
	50°	0.6	42.3	20.7	21.6	51	5.7	1.3	7.0
	60°	1.0	43.9	21.3	22.6	51	5.8	0.8	6.6
	70°	1.1	45.3	21.6	23.7	52	5.8	1.0	6.8
	80°	1.6	46.6	22.2	24.4	52	5.6	1.4	7.0
	90°	2.7	48.8	24.0	24.8	51	5.4	2.0	7.4
	100°	3.3	50.9	26.0	24.9	49	4.3	3.1	7.4
T3	2°	0.0	29.8	18.3	11.5	39	2.4	5.2	7.6
	10°	0.2	35.3	18.6	16.7	47	2.7	5.0	7.7
	20°	0.3	38.9	18.4	20.5	53	3.2	4.4	7.6
	30°	0.3	39.6	18.8	20.8	53	4.1	3.7	7.8
	40°	0.3	39.6	18.8	20.8	53	4.4	3.4	7.8
	50°	0.8	38.8	18.9	19.9	51	5.2	2.7	7.9
	60°	0.8	39.8	19.2	20.6	52	5.5	2.0	7.5
	70°	1.2	40.8	19.4	21.4	52	5.6	2.2	7.8
	80°	1.6	41.8	19.6	22.2	53	5.2	2.6	7.8
	90°	2.3	43.5	20.7	22.8	52	4.6	3.6	8.2
	100°	3.0	45.5	21.9	23.6	52	3.3	4.9	8.2

46 grams of wet hide powder was used for detannization instead of an amount adjusted to the tannin concentration, but these errors are believed to be too small to influence the results. The tannin concentration was less than 3.75 grams per liter for *Rhus copallina* extracts obtained at 20° to 60° C., inclusive, and *R. glabra* extracts obtained at 50° and 60° and for *R. typhina* extracts obtained at 20° and 50° C. The concentration was between 4.25 and 4.40 grams tannin per liter for *R. coriaria* and *R. glabra* extracts obtained at 100° C.

Another factor that might influence the results was that all extracts obtained by leaching at 80° C., or below, were heated to 80° at the end of the 7-hour leaching period, as specified in the Official method. The question might arise as to whether this heating had caused a change in composition of those extracts obtained at the low temperatures. In Table VI are given analyses of solutions obtained by making duplicate extractions at 20° C. of eight of the samples, heating one extract to 80° C. before analysis, and analyzing the duplicate portion without heating. A small crystal of 2-chloro-5-hydroxytoluene was added to the unheated solutions to check fermentation. No difference in insolubles in extractive or non tannin is evident from the data. There is a suggestion that heating to 80° C. may result in a slight loss of tannin and soluble solids, but this seems questionable since insolubles do not increase.

TABLE VI
EFFECT OF HEATING EXTRACTIVE TO 80° C. BEFORE ANALYSIS.
EXTRACTIVE SOLUTIONS OBTAINED BY LEACHING SUMAC AT 20° C.

Sample No.	Treatment	Insolubles in Extractive	Soluble Solids	Non Tannin	Tannin
		%	%	%	%
C2	heated.....	0.2	38.8	16.1	22.7
	not heated.....	0.3	39.2	16.0	23.2
C3	heated.....	0.2	37.2	17.6	19.6
	not heated.....	0.2	37.9	17.9	20.0
S1	heated.....	0.5	48.8	17.9	30.9
	not heated.....	0.7	48.2	17.3	30.9
S3	heated.....	0.3	46.1	17.8	28.3
	not heated.....	0.4	46.4	17.8	28.6
G1	heated.....	0.1	42.5	17.5	25.0
	not heated.....	0.1	42.7	17.4	25.3
G3	heated.....	0.3	44.1	21.2	22.9
	not heated.....	0.7	44.0	21.0	23.0
T1	heated.....	0.8	43.5	19.4	24.1
	not heated.....	0.8	44.8	19.3	25.5
T3	heated.....	0.3	38.9	18.4	20.5
	not heated.....	0.4	38.7	18.2	20.5

Tannin, as may be noted from Tables II to V, was quite low at 2° C., rose rapidly until the extraction temperature reached 30° or 40° C., then decreased as the extraction temperature rose to about 50° C., after which it increased steadily as the temperature was increased to 100° C.; the highest temperature studied.

The minimum point was at 50° C. for *R. glabra* and *R. typhina*, but it was a few degrees higher than 50° C. for *R. copallina* and a few degrees lower than 50° C. for *R. coriaria*. This behavior is easily observed in Figure I.

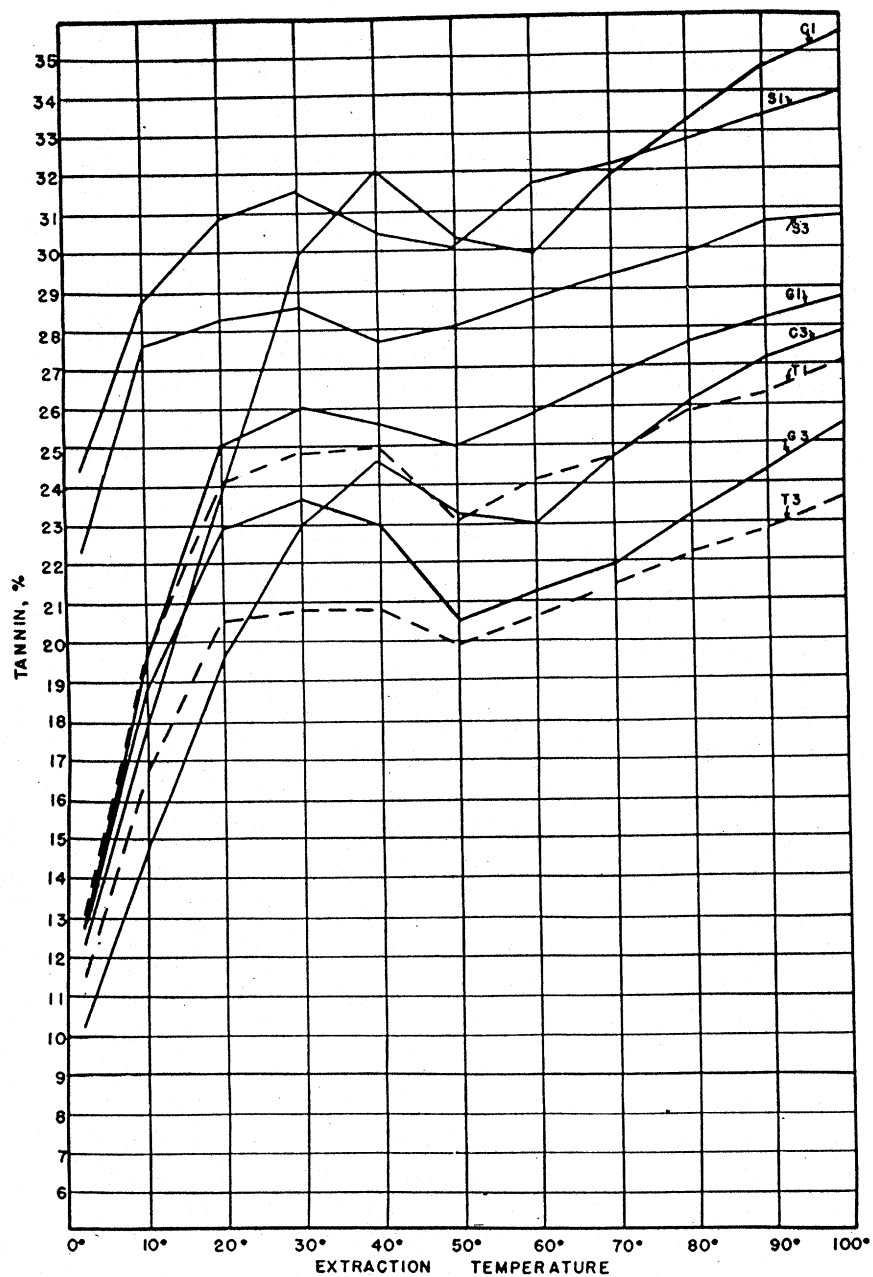


FIGURE I. Effect of extraction temperature on amount of tannin leached from sumac leaves. The symbols (C1, S1, etc.) are sample designations; see description of samples in text.

Although a relatively large amount of tannin was extracted at or near 30° C., yet without exception more tannin was found at 100° C. than at the lower temperature.

The optimum temperature for the extraction of sumac, if maximum tannin yield is desired, is thus shown to be 100° C.

In Figure I the two curves for *Rhus coriaria* are much higher at 2° C. than those for the remaining species. This difference will be discussed later.

Non tannin, as may be noted in Figure II, increased only slightly as the temperature of extraction was raised from 10° to 80° C., but there were more pronounced increases between 2° and 10° and above 80° C. The four species behaved in essentially the same manner. In previous work,⁷ a minimum in the curve showing variation of *Rhus glabra* non tannin with temperature of steeping was found at 70° C. No unusual behavior of *R. glabra* non tannin was noted in the present work.

Soluble solids showed nearly the same type of variability as tannin, since there was relatively little variation in non tannin. The amount of soluble matter extracted at 100° C. was greater than the maximum amount extracted at 30° or 40° C. by about 13 per cent for *Rhus copallina* and *R. glabra*, by about 10 per cent for *R. coriaria*, and by nearly 17 per cent for *R. typhina*. These percentages are based on the amount extracted at the lower temperature.

Purity of extractive was low at the low temperatures, but at 30° C. or above was nearly constant for most samples.

Reducing sugars in *Rhus typhina* were relatively high in extracts obtained at 50° to 70° C. but decreased considerably as the extraction temperature was either increased or decreased from this point. This same type of variability was shown by the two *R. glabra* samples collected in 1944 (G2 and G3) and the *R. copallina* sample collected in 1945 (C1). The remaining samples, which were harvested before 1944, all showed much less variability.

Total sugars were much less variable than reducing sugars. There was an increase in amount at the higher temperatures, but below 60° or 70° C. the amount extracted was essentially constant. Sugars in the more recently collected samples showed a tendency, although a very slight one, to vary in the same manner as soluble solids.

Determinations were made of pH values of the soluble solids solutions obtained by leaching at 2°, 10°, 20°, 30°, 60°, 90°, and 100° C. The greatest difference between any two of the 84 values was only 0.7. The average pH value for *Rhus copallina* was 3.99, for *R. coriaria* and *R. glabra*, 4.19 and for *R. typhina*, 4.24. Below 30° C. the average pH was slightly (about 0.2) but significantly greater than it was at or above this temperature.

As was mentioned previously, there was a noticeable difference between *Rhus coriaria* and the three domestic species in the amount of soluble matter

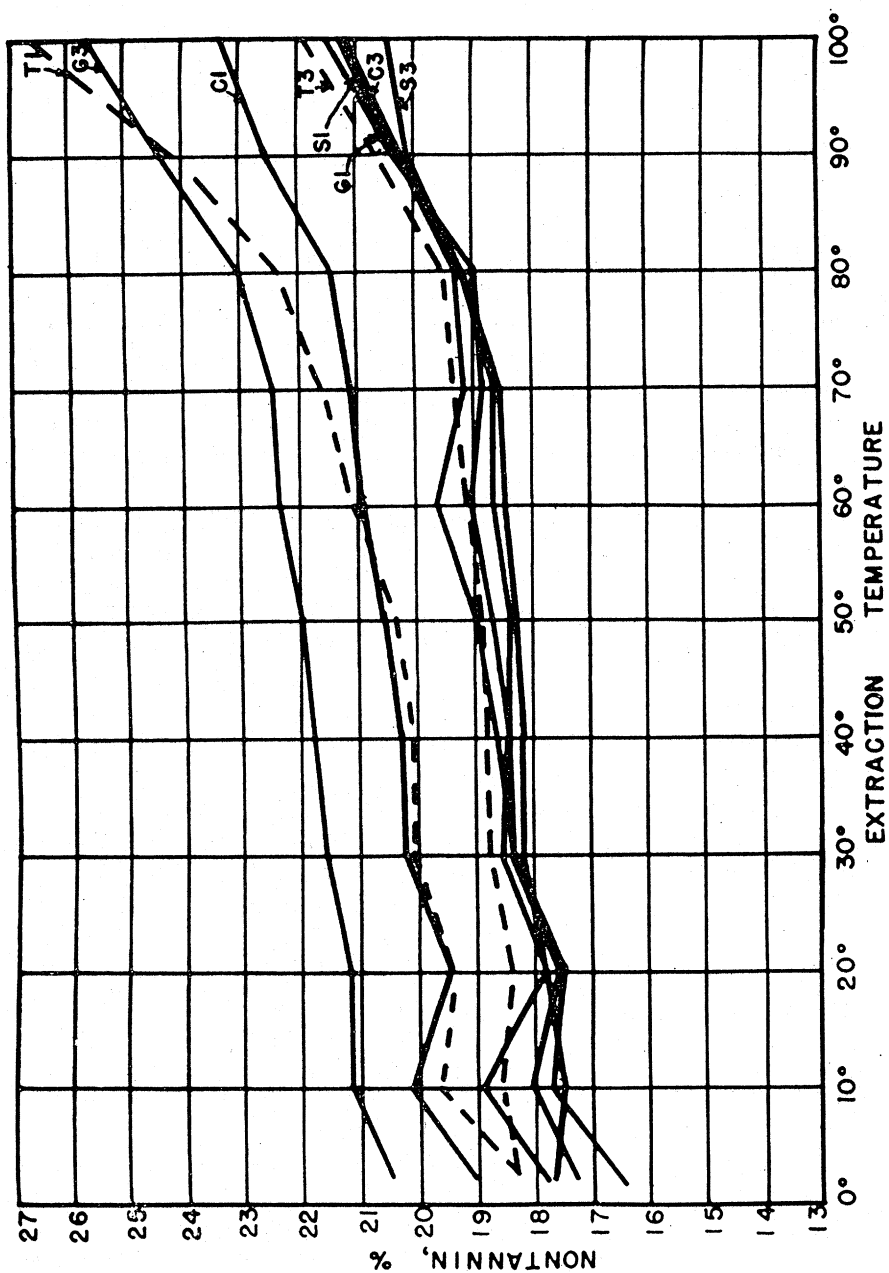


FIGURE II. Effect of extraction temperature on amount of non tannin leached from sumac leaves. The symbols (T1, G3, etc.) are sample designations; see description of samples in text.

TABLE VII
FINENESS OF GRIND OF SUMAC SAMPLES AS INDICATED BY SIEVING TESTS

Sample No.*	Proportion that Passed U. S. Standard Sieve Number					
	60	80	100	140	200	325
	%	%	%	%	%	%
C1	14.7	8.9	6.9	4.8	3.0	1.5
C1f	84.4	69.3	59.1	48.0	23.5	2.0
S4	24.5	14.7	11.4	8.6	5.8	3.6
S4f	92.8	81.2	69.4	53.6	12.5	0.8
G1	11.6	6.4	4.6	2.6	1.1	0.2
G1f	78.8	60.0	50.0	38.4	8.8	0.6
T1	21.6	9.6	6.3	3.5	1.9	0.7
T1f	93.8	78.0	61.6	37.1	4.0	0.4
S2	55.2	40.4	33.9	26.1	20.5	14.5
S1	54.2	36.7	31.9	26.1	24.4	13.9

*Samples C1, S4, G1, and T1 were ground in a Wiley mill to pass a 2 mm. (equivalent to U. S. No. 10 sieve) screen. C1f, S4f, G1f, and T1f first were ground to pass a 2 mm. screen then milled for 30 minutes in a pebble mill.

extracted at 2° C., as compared with the amounts extracted at 30° C. Aside from possible differences that might be ascribed to the nature of the species, one of the most evident differences between the imported and domestic samples was in fineness of grind. To determine whether fineness would explain the difference in leaching behavior, portions of samples C1, G1 and T1 were milled for 30 minutes in a pebble mill (producing samples C1f, G1f and T1f) then leached as before at low temperatures. A quantity of *R. coriaria* leaf

TABLE VIII
EFFECT OF FINENESS OF GRIND ON EXTRACTION OF SUMAC AT LOW TEMPERATURES

Sample Nos.	Extraction Temperature	Insolubles Sample Preparation*		Soluble Solids Sample Preparation*		Tannin Sample Preparation*	
		Coarse	Fine	Coarse	Fine	Coarse	Fine
	° C.	%	%	%	%	%	%
C1 and C1f (<i>R. copallina</i>)	2°	0.2	0.2	31.4	40.8	12.3	21.1
	20°	0.1	0.4	43.1	52.2	23.6	32.2
	30°	0.3	0.6	50.2	52.3	29.9	32.0
	40°	0.3	0.6	52.3	52.6	32.0	32.2
S4 and S4f (<i>R. coriaria</i>)	2°	0.3	0.2	29.3	37.6	13.1	20.2
	20°	0.4	0.8	43.0	44.3	25.5	26.4
	30°	0.3	0.6	44.8	44.3	26.8	26.0
	40°	0.4	1.2	44.8	44.8	26.8	26.4
G1 and G1f (<i>R. glabra</i>)	2°	0.3	0.2	29.3	40.6	12.8	21.9
	20°	0.1	0.8	42.5	43.8	25.0	25.9
	30°	0.1	1.1	44.3	43.6	26.0	25.3
	40°	0.4	0.4	44.0	43.3	25.6	25.0
T1 and T1f (<i>R. typhina</i>)	2°	0.3	0.7	31.2	40.9	13.0	21.0
	20°	0.8	1.2	43.5	44.0	24.1	24.2

*The coarse samples were C1, S4, G1, and T1; the fine were C1f, S4f, G1f and T1f. See footnote 1, Table VII.

was ground in a Wiley mill to pass a 2 mm. screen (sample S4), and an additional quantity was first ground to pass a 2 mm. screen then milled in a pebble mill as for the domestic species (sample S4f).

The results of sieving tests on these samples are given in Table VII and analytical data are given in Table VIII. The finely ground material was extremely difficult to leach; however, enough data were obtained to show clearly that with respect to leaching efficiency at low temperatures the difference between the domestic and imported sumacs was due to fineness of material and not to any inherent differences in species.

The data also show that the effect of particle size on leaching efficiency, although pronounced at 2° C., was almost absent at 20° C. or above for all species except *Rhus copallina*. For the latter species, the coarser material was leached less completely than the finer below 40° C.

Some data on rate and completeness of extraction at low temperatures are given in Table IX. Extractions were made for seven-hour periods at 2° C. and also at 10° C., followed first by a second seven-hour period at 2° or 10° C., respectively, and then by a third seven-hour period at 100° C. To condense the data, only averages with their standard deviations have been given, but the individual results for each sample for the first extraction period will be found in Tables II to V, inclusive. For tannin, separate averages have been calculated for the nine domestic sumac samples and for the three *Rhus coriaria* samples. Although less than half the tannin in the domestic sumacs was removed in seven hours at 2° C., a second seven-hour period removed only about one-fourth the remaining tannin. A number of seven-hour periods would have been required to remove all the tannin at this temperature, assuming that all could eventually have been removed. In almost every instance, more tannin was removed by one seven-hour extraction at 100° C. than by starting at either 2° or 10° C. and then finishing at 100° C., even though a total volume of 6 liters was obtained in the latter case and only 2 liters in the former.

Another type of extraction was made by starting at 20° C. and raising the temperature in 10° steps every 45 minutes until 100° C. was reached, collecting 220 ml. portions at each temperature. Such a type of extraction is often recommended for leaching tanning materials. The results, given in the last line in Table IX, are lower than those for the extractions in which the temperature was kept at 100° C. throughout the seven-hour period. Apparently leaching at 100° C. is preferable to any other method if maximum tannin yield is desired.

The non tannin values were little affected by temperature, but, as would be expected, more non tannin was removed when a total of 6 liters (3 periods) was used than when only 2 liters was used.

The total amount of insolubles in extractive (Table IX) was greater if the

TABLE IX
EFFICIENCY OF LOW TEMPERATURE EXTRACTION AND OF EXTRACTION
WITH A PROGRESSIVE TEMPERATURE RISE

Extraction Method*	Average† and Standard Deviation			
	Insolubles in Extractive	Non Tannin	Tannin	
			Domestic	<i>Rhus coriaria</i>
	%	%	%	%
One period at 100° C.‡....	3.11 ± 0.47	22.55 ± 2.34	28.24 ± 3.90	32.03 ± 1.71
First period at 2° C.‡....	0.21 ± 0.13	17.91 ± 1.49	11.77 ± 1.22	22.83 ± 1.27
Second period at 2° C....	0.13 ± 0.06	1.55 ± 0.74	3.91 ± 0.97	2.93 ± 0.25
Third period at 100° C....	2.23 ± 0.30	4.58 ± 0.36	11.03 ± 4.26	5.97 ± 0.41
Total.....	2.57 ± 0.30	24.04 ± 1.32	26.71 ± 3.57	31.73 ± 1.66
First period at 10° C.‡....	0.27 ± 0.08	18.68 ± 1.40	18.01 ± 1.74	28.30 ± 0.62
Second period at 10° C....	0.17 ± 0.15	1.27 ± 0.31	4.26 ± 1.54	1.13 ± 0.15
Third period at 100° C....	1.59 ± 0.17	3.69 ± 0.27	5.58 ± 2.16	3.20 ± 0.36
Total.....	2.03 ± 0.23	23.64 ± 1.29	27.84 ± 3.20	32.63 ± 1.06
First period at 20° C.‡....	0.42 ± 0.24	18.97 ± 1.41	22.23 ± 0.19	29.43 ± 1.47
Second Period at 100° C...	1.80 ± 0.23	4.55 ± 0.43	5.88 ± 3.17	3.03 ± 0.15
Total.....	2.22 ± 0.32	23.52 ± 1.62	28.11 ± 3.57	32.47 ± 1.63
One period 20° C. to 100° C.	1.24 ± 0.24	20.31 ± 1.41	27.51 ± 3.66	31.63 ± 1.57

*Each period consisted of one 7-hour extraction during which 2 liters of percolate were collected.

†Averages for insolubles in extractive and non tannin are for all 12 samples; domestic tannin averages are for samples C1, C2, C3, G1, G2, G3, T1, T2, and T3; *Rhus coriaria* averages are for samples S1, S2 and S3.

‡Individual values for each sample will be found in Tables II to V inclusive.

TABLE X
EFFECT OF EXTRACTION TEMPERATURE ON COLOR OF SOLUBLE SOLIDS
SOLUTIONS AS INDICATED BY TRANSMISSION OF BLUE LIGHT

Sample No.	Transmission of Blue Light (436 mμ)* Extraction Temperature ° C.						
	30°	40°	50°	60°	70°	80°	90°
	%	%	%	%	%	%	%
C1.....	82.6	81.2	81.0	80.2	79.2	77.7	72.0
C2.....	66.1	62.6	62.4	56.4	59.3	57.8	57.4
C3.....	53.0	48.2	47.7	47.5	45.2	45.7	46.0
S1.....	72.4	72.1	73.0	71.2	68.4	61.4	64.0
S2.....	69.0	68.2	68.8	68.4	65.8	61.3	60.5
S3.....	67.8	66.2	67.0	65.3	63.8	60.3	56.3
G1.....	42.2	38.7	39.5	39.3	37.8	37.3	40.7
G2.....	73.6	72.7	72.3	70.9	69.3	69.3	65.1
G3.....	74.2	72.3	73.0	72.2	71.6	68.3	62.0
T1.....	70.3	68.1	68.0	68.2	65.3	63.3	55.4
T2.....	72.6	71.1	70.4	70.8	66.7	67.7	53.2
T3.....	72.1	69.8	68.7	68.4	61.9	55.7

*Compared with distilled water. Cell thickness 5 mm.

extraction was made entirely at 100° C. than if much of the soluble matter was first removed at a low temperature before completing the extraction at 100° C. This seems to indicate that high insolubles are the result of higher temperatures.

Color measurements were made on some of the soluble solids solutions. The transmission values for blue light (436 millimicrons) given in Table X decrease somewhat as the extraction temperature was raised from 30° to 90° C., that is, the solutions obtained at the higher temperatures were darkest. Sheepskin skivers tanned with total solids solutions, however, showed no consistent trend in color values as the extraction temperature was raised, as may be noted in Table XI. Frequently the skivers tanned with extracts obtained at 90° C. were lighter in color than skivers tanned with extracts obtained at 30° or 60° C.

TABLE XI

EFFECT OF EXTRACTION TEMPERATURE ON COLOR* OF TANNED SHEEPSKIN SKIVERS

Sample No.	Reflectance of Green Light Extraction Temperature ° C.			Reflectance of Blue Light Extraction Temperature ° C.		
	30°	60°	90°	30°	60°	90°
	%	%	%	%	%	%
C1.....	68.4	65.7	68.1	56.4	53.2	55.7
C2.....	49.9	47.7	60.5	35.4	35.1	44.2
C3.....	51.8	53.0	56.5	35.3	36.5	40.6
S1.....	58.1	63.7	66.8	46.6	51.3	54.2
S2.....	61.5	65.5	62.5	48.4	51.2	49.3
S3.....	62.7	66.0	63.8	49.6	51.4	50.4
G1.....	50.9	57.0	54.6	34.5	39.0	38.3
G2.....	62.8	64.9	68.5	51.1	51.9	55.7
G3.....	62.6	58.5	65.3	53.8	49.5	54.7
T1.....	61.5	62.3	61.5	50.6	50.3	49.6
T2.....	64.3	62.7	61.3	54.2	51.8	51.7
T3.....	61.3	66.3	65.9	49.8	52.2	51.0

*Percentage reflectance for green light (546 millimicrons) and for blue light (436 millimicrons) of sheepskin skivers tanned with total solids solutions. Reflectance versus MgO, 45° incidence, normal viewing.

Discussion

The eight curves in Figure I showing variation of tannin with extraction temperature have approximately the same general shape. They rise from 2° to 100° C., except for a dip, corresponding to decreased extraction of tannin, with a minimum point in the neighborhood of 60° C. At present the reason for this dip is not known. The curve rises at 100° C., indicating that the maximum point is above this temperature. This agrees with the results of Eitner,³ which indicated that there was maximum extraction of tannin at some temperature above 100° C.

TABLE XII
PARKER AND PROCTER'S* RESULTS ON EFFECT OF
TEMPERATURE IN EXTRACTION OF SUMAC

Extraction Temperature ° C.	Soluble Solids† %	Non Tannin %	Tannin %	Purity of Extractive†	Color of Soluble Solids Lovibond System	
					Red	Yellow
15°.....	32.0	17.8	14.2	44	1.6	5.4
15°- 30°.....	35.7	18.1	17.6	49	1.4	4.3
30°- 40°.....	36.6	18.1	18.5	51	1.3	4.4
40°- 50°.....	38.6	18.5	20.1	52	1.4	4.4
50°- 60°.....	39.4	19.1	20.3	52	1.5	4.7
60°- 70°.....	38.4	19.4	19.0	49	1.7	5.6
70°- 80°.....	37.9	19.9	18.0	47	1.9	6.2
80°- 90°.....	38.0	21.1	16.9	44	2.3	6.8
90°-100°.....	38.9	22.3	16.6	43	2.6	7.0
Boiled 0.5 hour.....	39.2	24.0	15.2	39	3.3	7.7

*See reference 9.

†Calculated from Parker and Procter's values for non tannin and tannin.

Our tannin data are not in agreement with those of Parker and Procter,⁸ given in Table XII. Their results did not agree with other work of that period for they state that: "In this experiment the maximum yield of tanning matter was at 50° to 60° C. while Seymour-Jones, and Palmer who first drew attention to the matter found it between 30° and 40° C., a result confirmed by J. T. Wood."* They also stated that the matter needed further inquiry and that the increase in non tannin corresponding to the diminished yield of tannin at higher temperatures indicated some decomposition. The highest purity of extractive obtained by Parker and Procter (Table XII) was 52 at 50° to 60° C., and purity decreased rapidly as the temperature rose above 60° C. The purities of our extractives were all much higher than theirs and showed no decrease at higher temperatures. This would seem to confirm their opinion that there was a destruction of tannin by their method of leaching, but the cause can not now be determined. An attempt to check their results by making an extraction at 90° C. according to their directions was not successful; we did not obtain a comparable loss of tannin. In previous work⁷ we found that tannin apparently was changed to non tannin on steeping at elevated temperatures. Parker and Procter soaked their sumac overnight at room temperature, then heated it in a water bath to the desired temperature of extraction. Such a mild steeping should not cause the large loss of tannin they found. Their non tannin values agree very well with our values, and the lack of agreement in soluble solids values and also the darker color of their soluble solids solutions at higher temperatures would be explained by a destruction of tannin.

* We were unable to locate the original articles by Seymour-Jones, Palmer and Wood.

Sugar was easily extracted from sumac even at 2° C., as was noted above in Tables II to V. Above 80° C. there was an increase in total sugar due to the production or liberation of some compound that would reduce Fehling's solution. Although the amount of total sugar was nearly constant regardless of extraction temperature, reducing sugar was variable. As the extraction temperature rose from 2° to 60° or 70° C., there was an increase in reducing sugar and a corresponding decrease in nonreducing sugar, indicating hydrolysis of the nonreducing sugar during the extraction. Above 70° C., reducing sugar decreased with increase in extraction temperature. This decrease may have been caused by destruction of sugar at the higher temperature, or if hydrolysis below 70° C. was caused by enzymes, it may have been due to inactivation of the enzymes. This rise in reducing sugar to a maximum at about 70° C. was very pronounced for the most recently collected samples, but it was still in evidence for the older samples.

As a whole, these results, insofar as they are applicable to commercial practice, indicate that temperatures of 100° C. should be used for the commercial leaching of sumac. At 100° C. the greatest yield of tannin should be obtained, and purity of the extract should be practically as high as of extracts prepared at lower temperatures. Color tests were not made on extracts obtained at 100° C., but color skivers tanned with extracts leached at 90° C. were not in general darker than those tanned with extracts obtained at lower temperatures.

Summary

Three samples each of *Rhus copallina*, *R. glabra*, *R. typhina*, and *R. coriaria* were extracted in a modified Reed-Churchill extractor, and leached so as to obtain two liters of extractive in seven hours. Throughout the extraction period there was a deviation from the desired temperature of less than 0.1 degree at 30° C. and above, while the deviation for the lower temperature was not more than 0.5 degree. As the temperature of extraction was increased, the amount of tannin and soluble matter rose from a low of 2° to a high point at 30° to 40° C., decreased to a point between 40° and 60° C., and then rose steadily to a maximum of 100° C. More tannin was removed by maintaining the temperature at 100° C. than by starting at a lower temperature and raising it during the extraction.

Finely divided material leached more completely at low temperatures than coarse material, but at 30° C. or above there was little difference in this respect. There were relatively small fluctuations in sugars and pH values.

Transmission measurements showed some darkening of the soluble solids solutions as the temperature of extraction was raised from 30° to 90° C. Skivers tanned with the total solids solutions showed no differences in color that could be attributed definitely to effect of extraction temperature.

Acknowledgment

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Discussion

L. SHEARD: Mr. Chairman, this has been a most interesting paper, not only because it compared the domestic sumacs with the Sicilian, but because it has cleared up to some extent, at least so far as sumac is concerned, the low temperatures which were presumed to be best for extracting the maximum amount of tannin. Researches by Parker, Procter, Jones, Palmer, and Wood showed, as has been stated by the authors of this paper, that the optimum temperature for Sicilian sumac was below 60° C. All of these gentlemen were eminent in research in the field of leather chemistry and I wonder if the author can explain the difference in the two conclusions.

Another interesting point mentioned in the paper was that more tannin was removed by one seven-hour period at 100° C. than by starting at 2° or 10° C. and finishing at 100° C., although six liters were obtained in the latter case and only two when the extraction was carried out at 100° C. during the whole period.

I think, Mr. Chairman, this has been an excellent paper and a valuable paper from the Eastern Regional Research Laboratory.

I. D. CLARKE: We are not able to explain the difference between our results and those of Parker and Procter, nor the reason that the two liters gives more tannin than the six liters. But it is not true for the non tannin. It is only the tannin. The non tannin gives the expected difference. Six liters give more non tannin.